Report:	IIA 6.1.1/02	Cavaillé, C.; Meilland-Berthier, I.; (2011). Storage stability of residues of BYF 14182 and its metabolites (BCS-AA10006, BCS-AA10790, BCS-AA10791 and BCS-CM41431) in plants during deep freeze storage for up to 24 months. Bayer CropScience AG. Report no. MR-09/136. 17th June 2011. Unpublished. MRID No. 48516101
Guidelines:		OECD Guideline No. 506: Stability of pesticide residues in stored commodities; PMRA Residue Chemistry Guidelines DIR98-02: Section 5 Storage Stability Data; US EPA OCSPP 860.1380 – Storage Stability Data
GLP:	Yes	OECD Principles of GLP and French decree n° 2006-1523 from 04/12/2006
GLP Exceptions:	No	
Acceptability:		This study is considered to be acceptable.

Executive Summary

A study was conducted to investigate the storage stability of BYF 14182 and its metabolites BYF 14182-hydroxy-butyl, BYF 14182-pyrazole-4-carboxamide, BYF 14182-homoglutathione or BYF 14182-bis-desmethyl-3-carboxylic acid in matrices of plant origin when stored under freezer conditions. Samples of potato (tuber), lettuce (head), orange (fruit), dry bean (seed), wheat (grain and straw) and sunflower (seed) were fortified with the test items at 0.20 mg/kg (1 mg/kg for wheat straw). Samples were stored at ≤-18°C and were analysed after storage intervals of 0, 3 – 4, 8 – 9, 16 – 17 and 26 – 27 months.

At each storage interval levels BYF 14182 and its metabolites were determined using analytical method 01057 which involves extraction with acetonitrile/water. After hydrolysis with 6 N HCl, the extract is neutralized with NaOH before analysis by LC/MS/MS with internal standards for BYF 14182 (parent), BYF 14182-hydroxy-butyl, BYF 14182-pyrazole-4-carboxamide, with internal or external standards for BYF 14182-homoglutathione and external standards for BYF 14182-bis-desmethyl-3-carboxylic acid. The overall means of the concurrent recoveries for all matrices and analytes were within the range of 70 – 110% with the exception of the concurrent recovery of BYF 14182 from sunflower seed which had a mean recovery of 62%. Individual concurrent recoveries were variable for some matrices and outside the acceptable range in several instances. Residues of BYF 14182 and its metabolites in unfortified control samples were generally below the Limit of Quantification (<0.01 mg/kg or <0.05 mg/kg for wheat straw) with the exception of a dry bean sample and a wheat straw sample which contained contained BYF 14182-homoglutathione at the LOQ (These results were reported to be due to contamination of the control samples during preparation or due to contamination of the dilution solvent used in the method).

Possible degradation of parent and BYF 14182-homoglutathione in dry bean were noted in the initial evaluation of the study for up to 9 months storage, however the 17 month interim report appeared to show acceptable recoveries at this time point. This is confirmed now that the final study is available. Issues with storage stability recoveries of parent from sunflower seed appear to be related to problems with the method. The storage stability of parent in sunflower seed is acceptable if corrected for concurrent recoveries.

After a deep-freezer storage period of 26 – 27 months mean corrected recovery values ranged from 87 – 109% for BYF 14182, 90 – 103% for BYF 14182-hydroxy-butyl, 85 – 123% for BYF 14182-homoglutathione, 92 – 105% for BYF 14182-pyrazole-4-carboxamide and 100 – 114% for BYF 14182-bis-desmethyl-3-carboxylic acid. BYF 14182 and its metabolites are considered to be stable for at least 26 months in potato (tuber), lettuce (head), dry bean (seed), orange (fruit), wheat (grain and straw) and sunflower (seed) when stored frozen at ≤-18°C.

I. MATERIALS AND METHODS

A. Materials

1. Test Materials

BYF-14182

Certificate of Analysis	AZ 15302, 2008-09-26 (1) AZ 15705, 2009-04-06 (2)
Chemical Name	N-[2-(1,3-dimethylbutyl)phenyl]-5-fluoro-1,3-dimethyl-1H-pyrazole-4-carboxamide
Empirical Formula	$C_{18}H_{24}FN_3O$
Molar Mass	317.41 g/mol
Purity	99.5 % (1) 99.2 % (2)
Expiry Date	2011-09-17 (1) 2012-03-31 (2)

BYF 14182-3-hydroxy-butyl

Code number	BCS-AA10006
Certificate of Analysis	AZ 15083, 2008-06-25 (1) AZ 16681, 2010-06-14 (2)
Chemical Name	5-fluoro-N-[2-(3-hydroxy-1,3-dimethylbutyl)phenyl]-1,3-dimethyl-1H-pyrazole-4-carboxamide
Empirical Formula	$C_{18}H_{24}FN_3O_2$
Molar Mass	333.41 g/mol
Purity	99.2 %
Expiry Date	2010-06-20 (1) 2013-05-25 (2)

BYF 14182-homoglutathione

Code number	BCS-AA10790
Certificate of Analysis	AZ 14961, 2008-03-31 (1) AZ 15423, 2008-11-11 (2) AZ 16973, 2010-11-08 (3)
Chemical Name	Gamma-glutamyl-S-(4-{[2-(1,3-dimethylbutyl)phenyl]carbamoyl}-1,3-dimethyl-1H-pyrazol-5-yl)cysteinyl-beta-alanine
Empirical Formula	$C_{29}H_{42}N_6O_7S$
Molar Mass	618.75 g/mol
Purity	99.0 % (1)

	87.0 % (2)
	85.0 % (3)
Expiry Date	2010-03-27 (1)
	2010-11-04 (2)
	2012-10-29 (3)

BYF 14182-pyrazole-4-carboxamide

Code number	BCS-AA10791
Certificate of Analysis	AZ 14748, 2008-01-18 (1)
	AZ 16367, 2010-01-14 (2)
Chemical Name	5-fluoro-1,3-dimethyl-1H-pyrazole-4-carboxamide
Empirical Formula	$C_6H_8FN_3O$
Molar Mass	157.15 g/mol
Purity	98.6 %
Expiry Date	2010-01-14 (1)
	2013-01-08 (2)

BYF 14182-bis-desmethyl-3-carboxylic acid

Code number	BCS-CM41431
Certificate of Analysis	AZ 15553, 2008-12-11 (1)
	AZ 17017, 2010-11-18 (2)
Chemical Name	5-fluoro-1H-pyrazole-3-carboxylic acid
Empirical Formula	$C_4H_3FN_2O_2$
Molar Mass	130.08 g/mol
Purity	> 99.9 % (1)
	97.4 % (2)
Expiry Date	2010-12-03 (1)
	2012-11-16 (2)

2. Test Commodity

The control material used for fortification was acquired in the market on 2008-10-18 or 2008-10-20 or taken from the Bayer sample logistic in Monheim (PVTL-Bayer CropScience). Details are summarised in Table B.7.6.2-6.

Table B.7.6.2-6 Origin of control samples used in study.

Sample Material	Origin of Control Material	Storage
Potato (tuber)	Bought in the shop (SATORIZ in Champagne au Mont d'Or) (France)	At or below around -18 °C
Lettuce (head)	Bought in the shop (SATORIZ in Champagne au Mont d'Or) (France)	At or below around -18 °C
Wheat (grain)	From residue study RA-2066/07 trial 0835/1 Monheim (PVTL-Bayer CropScience)	At or below around -18 °C
Wheat (straw)	From residue study RA-2066/07 trial 0835/1 Monheim (PVTL-Bayer CropScience)	At or below around -18 °C
Dry bean (seed)	Bought in the shop (SATORIZ in Champagne au Mont d'Or) (France)	At or below around -18 °C
Sunflower (seed)	Bought in the shop (SATORIZ in Champagne au Mont d'Or) (France)	At or below around -18 °C

Orange (fruit)	Bought in the market in Villeurbanne (France)	At or below around -18 °C

B. Study design

The study was conducted between November 2008 and June 2011 with stability of residues determined after 26 - 27 months storage. Each sample material was fortified individually at 0.2 mg/kg (1 mg/kg for wheat straw) with parent compound (BYF 14182), BYF 14182-hydroxy-butyl, BYF 14182-pyrazole-4-carboxamide, BYF 14182-homoglutathione or BYF 14182-bis-desmethyl-3-carboxylic acid. Samples were stored in sealed plastic bottles at ≤-18°C. Residues of BYF 14182 and its metabolites in the samples were determined after the nominal storage intervals of 0, 3, 9, 16 and 24 months by the analytical method 01057. Samples were extracted twice with a mixture of acetonitrile/water (80:20 v/v). Extracts were hydrolysed with 6N HCl at 75°C for 15 minutes (this step hydrolyses conjugates of BYF 14182-hydroxy-butyl). The extract was neutralized with 6 N NaOH and an aliquot diluted prior to quantification by LC/MS/MS with internal standards for BYF 14182 (parent), BYF 14182-hydroxy-butyl, BYF 14182-pyrazole-4-carboxamide, internal or external standards for BYF 14182-homoglutathione and external standards for BYF 14182-bis-desmethyl-3-carboxylic acid.

Method validation experiments were conducted prior to sample storage for sunflower seed only. Control samples of sunflower seed were fortified with BYF 14182 (parent), BYF 14182-hydroxy-butyl, BYF 14182-homoglutathione, BYF 14182-pyrazole-4-carboxamide and BYF 14182-bis-desmethyl-3-carboxylic acid at 0.01 and 0.1 mg/kg and then analysed. In addition, concurrent recovery experiments were performed for all samples at all storage intervals by fortifying stored control samples of each material with BYF 14182 and its metabolites at 0.01 and 0.20 mg/kg (0.05 and 1 mg/kg for wheat straw). At each storage interval, one stored control sample was analysed concurrently with the fortified samples.

II. RESULTS AND DISCUSSION

A summary of the recovery data generated for method validation for sunflower seed is given below in Table B.7.6.2-7.

Table B.7.6.2-7 Recovery data for method validation for BYF 14182 and its metabolites for sunflower seed.

Sample material	Analyte	Fortification level (mg/kg)	Recovery (%)	Mean (%)	RSD (%)
Sunflower (seed)	BYF 14182	0.01	73, 76, 67	72	6.4
		0.10	62, 70, 76	69	10.1
Sunflower (seed)	BYF 14182-hydroxy-butyl	0.01	96, 95, 88	93	4.7
		0.10	92, 89, 105	95	8.9
Sunflower (seed)	BYF 14182-homoglutathione	0.01	60, 66, 78	68	13.5
		0.10	98, 85, 114	99	14.7
Sunflower (seed)	BYF 14182-	0.01	95, 105, 79	93	14.1

	pyrazole-4-carboxamide	0.10	92, 96, 93	94	2.2
Sunflower (seed)	BYF 14182-bis-desmethyl-3-carboxylic acid	0.01	63, 91, 82	79	18.2
		0.10	82, 96, 81	86	9.7

Summaries of concurrent recoveries of BYF 14182 and its metabolites obtained from fortified control samples analysed at the same time as the test samples are given below in Tables B.7.6.2-8 to B.7.6.2-12. The overall means of the concurrent recoveries for all matrices and analytes were within the range of 70 – 110% with the exception of the concurrent recovery of BYF 14182 from sunflower seed which had a mean recovery of 62% (The variation in the recoveries for sunflower was reported to depend on the homogeneization of the raw extract due to the high fat content). Individual concurrent recoveries were variable for some matrices and outside the acceptable range in several instances.

Table B.7.6.2-8 Summary of concurrent recoveries of BYF 14182.

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182							
Potato (tuber)	0.01	0	1	90	-	-	-
		3	1	77	-	-	-
		9	1	97	-	-	-
		17	1	77	-	-	-
		26	1	78	-	-	-
	0.10	3	2	92 / 85	89	-	-
	0.20	0	3	91 / 81 / 77	83	8.7	7.2
		9	2	110 / 116	113	-	-
		17	2	95 / 67	81	-	-
		26	3	80 / 87 / 79	82	5.3	4.4
	Overall mean, RSD and Standard deviation				**87**	**14.4**	**12.5**
Lettuce (head)	0.01	0	1	94	-	-	-
		3	1	76	-	-	-
		9	1	82	-	-	-
		16	1	52	-	-	-
		26	1	70	-	-	-
	0.40	9	2	108 / 112	110	-	-
	0.20	0	3	90 / 85 / 83	86	4.2	3.6
		3	2	77 / 67	72	-	-
		16	2	63 / 67	65	-	-
		26	3	74 / 80 / 75	76	4.2	3.2
	Overall mean, RSD and Standard deviation				**80**	**19.2**	**15.3**
Dry bean (seed)	0.01	0	1	69	-	-	-
		3	1	86	-	-	-
		9	1	88	-	-	-
		17	1	80	-	-	-
		26	1	59	-	-	-
	0.20	0	3	63 / 64 / 59	62	4.3	2.6

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
		3	2	74 / 76	75	-	-
		9	2	114 / 107	111	-	-
		17	2	75 / 80	78	-	-
		26	3	85 / 64 / 65	71	16.6	11.8
	Overall mean, RSD and Standard deviation				**77**	**20.4**	**15.7**
Orange (fruit)	0.01	0	1	90	-	-	-
		3	1	80	-	-	-
		8	1	77	-	-	-
		16	1	83	-	-	-
		26	1	79	-	-	-
	0.20	0	3	89 / 90 / 87	89	1.7	1.5
		3	2	79 / 79	79	-	-
		8	2	92 / 101	97	-	-
		16	2	75 / 90	83	-	-
		26	3	68 / 82 / 76	75	9.4	7.0
	Overall mean, RSD and Standard deviation				**83**	**9.6**	**8.0**
Wheat (grain)	0.01	0	1	70	-	-	-
		4	1	73	-	-	-
		9	1	82	-	-	-
		16	1	81			
		26	1	79	-	-	-
	0.20	0	3	69 / 74 / 64	69	7.2	5.0
		4	2	62 / 68	65	-	-
		9	2	97 / 90	94	-	-
		17	2	90 / 73	82	-	-
		26	6	70 / 75 / 50 / 85 / 87 / 81	75	18.3	22.9
	Overall mean, RSD and Standard deviation				**75**	**15.3**	**11.4**
Wheat (straw)	0.05	0	1	84	-	-	-
		4	1	76	-	-	-
		8	1	92	-	-	-
		16	1	50	-	-	-
		26	1	62	-	-	-
	1	0	3	75 / 73 / 79	76	4.0	3.1
		4	2	81 / 77	79	-	-
		8	2	92 / 97	94	-	-
		16	4	62 / 60 / 51 / 51	56	10.9	5.8
		26	3	64 / 62 / 73	66	8.8	5.9
	Overall mean, RSD and Standard deviation				**72**	**19.8**	**14.2**
Sunflower (seed)	0.01	0	1	47	-	-	-
		4	1	64	-	-	-
		8	1	66	-	-	-
		16	1	53	-	-	-
		27		*			
	0.20	0	3	52 / 49 / 43	48	9.5	4.6
		4	2	67 / 65	66	-	-
		8	2	97 / 100	99	-	-
		17	2	68 / 41	55		
		27	3	61 / 46 / 72	60	21.7	13.1

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
	Overall mean, RSD and Standard deviation				**62**	**27.9**	**17.3**

*Result not provided as outside of the criteria.

Table B.7.6.2-9 Summary of concurrent recoveries of BYF 14182-hydroxy-butyl.

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-3-hydroxy-butyl							
Potato (tuber)	0.01	0	1	99	-	-	-
		3	1	95	-	-	-
		9	1	88	-	-	-
		17	1	76	-	-	-
		26	1	83	-	-	-
	0.10	3	2	83 / 91	87	-	-
	0.20	0	3	94 / 91 / 85	90	5.1	4.6
		9	2	87 / 94	91	-	-
		17	2	85 / 84	84	-	-
		26	3	85 / 89 / 86	87	2.4	2.1
	Overall mean, RSD and Standard deviation				**88**	**6.4**	**5.6**
Lettuce (head)	0.01	0	1	97	-	-	-
		3	1	92	-	-	-
		9	1	81	-	-	-
		16	1	89	-	-	-
		26	1	84	-	-	-
	0.40	9	2	86 / 96	91	-	-
	0.20	0	3	90 / 87 / 86	88	2.4	2.1
		3	2	78 / 77	78	-	-
		16	2	95 / 96	96	-	-
		26	3	86 / 91 / 88	88	2.8	2.5
	Overall mean, RSD and Standard deviation				**88**	**6.9**	**6.1**
Dry bean (seed)	0.01	0	1	84	-	-	-
		3	1	82	-	-	-
		9	1	106	-	-	-
		17	1	76	-	-	-
		26	1	75	-	-	-
	0.20	0	3	78 / 75 / 77	77	2.0	1.5
		3	2	65 / 67	66	-	-
		9	2	94 / 101	98	-	-
		17	2	74 / 78	76	-	-
		26	3	75 / 75 / 76	75	0.8	0.6
	Overall mean, RSD and Standard deviation				**80**	**13.7**	**10.9**
Orange (fruit)	0.01	0	1	85	-	-	-
		3	1	97	-	-	-
		8	1	74	-	-	-
		16	1	84	-	-	-
		26	1	75	-	-	-
	0.20	0	3	86 / 87 / 85	86	1.2	1.0
		3	2	79 / 109	94	-	-
		8	2	96 / 87	92	-	-

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-3-hydroxy-butyl							
		16	2	84 / 78	81	-	-
		26	3	86 / 81 / 90	85	5.1	4.5
	Overall mean, RSD and Standard deviation				**86**	**10.0**	**8.6**
Wheat (grain)	0.01	0	1	88	-	-	-
		4	1	86	-	-	-
		9	1	85	-	-	-
		16	1	98	-	-	-
		26	1	72	-	-	-
	0.20	0	3	88 / 71 / 82	80	10.7	8.6
		4	2	64 / 68	66	-	-
		9	2	99 / 90	95	-	-
		16	2	75 / 82	79	-	-
		26	3	80 / 80 / 76	79	2.9	2.3
	Overall mean, RSD and Standard deviation				**83**	**11.6**	**9.6**
Wheat (straw)	0.05	0	1	99	-	-	-
		4	1	87	-	-	-
		8	1	92	-	-	-
		16	1	83	-	-	-
		26	1	67	-	-	-
	1	0	3	87 / 89 / 86	87	1.7	1.5
		4	2	99 / 101	100	-	-
		8	2	78 / 78	78	-	-
		16	2	70 / 72	71	-	-
		26	3	73 / 76 / 76	75	2.3	1.7
	Overall mean, RSD and Standard deviation				**83**	**12.7**	**10.6**
Sunflower (seed)	0.01	0	1	82	-	-	-
		4	1	91	-	-	-
		8	1	81	-	-	-
		16	1	82	-	-	-
		26	1	71	-	-	-
	0.20	0	3	75 / 75 / 93	81	12.8	10.4
		4	2	87 / 85	86	-	-
		8	2	97 / 90	94	-	-
		16	2	79 / 85	82		
		26	3	89 / 81 / 77	83	7.0	6.1
	Overall mean, RSD and Standard deviation				**84**	**8.5**	**7.1**

Table B.7.6.2-10 Summary of concurrent recoveries of BYF 14182-homoglutathione.

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-homoglutathione							
Potato (tuber)	0.01	0	1	107	-	-	-
		3	1	98	-	-	-
		9	1	136	-	-	-
		17	1	107	-	-	-
		26	1	128	-	-	-
	0.10	3	2	105 / 109	107	-	-

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-homoglutathione							
	0.20	0	3	100 / 94 / 88	94	6.4	6.0
		9	2	128 / 132	130	-	-
		17	2	117 / 110	114	-	-
		26	3	123 / 134 / 105	121	12.1	14.6
	Overall mean, RSD and Standard deviation				**113**	**13.1**	**14.8**
Lettuce (head)	0.01	0	1	92	-	-	-
		3	1	74	-	-	-
		9	1	98	-	-	-
		16	1	110	-	-	-
		26	1	102	-	-	-
	0.40	9	2	116 / 120	118	-	-
	0.20	0	3	80 / 76 / 76	77	3.0	2.3
		3	2	91 / 89	90	-	-
		16	2	104 / 100	102	-	-
		26	3	97 / 91 / 127	105	18.4	19.3
	Overall mean, RSD and Standard deviation				**97**	**16.2**	**15.6**
Dry bean (seed)	0.01	0	1	64	-	-	-
		3	1	82	-	-	-
		9	1	77	-	-	-
		17	1	131	-	-	-
		26	1	94	-	-	-
	0.20	0	3	56 / 48 / 50	51	8.1	4.2
		3	2	106 / 105	106	-	-
		9	2	156 / 156 (**)	156	-	-
		17	2	111 / 98	104	-	-
		26	3	123 / 75 / 71	90	32.3	28.9
	Overall mean, RSD and Standard deviation				**94**	**35.8**	**33.7**
Orange (fruit)	0.01	0	1	83	-	-	-
		3	1	120	-	-	-
		8	1	88	-	-	-
		16	1	77	-	-	-
		26	1	104	-	-	-
	0.20	0	3	81 / 85 / 79	82	3.7	3.1
		3	2	76 / 99	88	-	-
		8	2	88 / 95	92	-	-
		16	2	87 / 97	92		
		26	3	105 / 93 / 75	91	16.9	15.1
	Overall mean, RSD and Standard deviation				**90**	**13.5**	**12.2**
Wheat (grain)	0.01	0	1	64	-	-	-
		4	1	79	-	-	-
		9	1	100	-	-	-
		16	1	119	-	-	-
		26	1	141	-	-	-
	0.20	0	3	71 / 70 / 109	83	26.7	22.2
		4	2	86 / 66	76	26.7	22.2
		9	2	79 / 84	82	-	-
		16	2	107 / 114	111	-	-
		26	6	98 / 120 / 61 /	87	23.4	20.3

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-homoglutathione							
				84 / 85 / 74			
	Overall mean, RSD and Standard deviation				**92**	**25.4**	**23.4**
Wheat (straw)	0.05	0	1	78	-	-	-
		4	1	80	-	-	-
		8	1	216 (***)	-	-	-
		16	1	123	-	-	-
		26	1	93	-	-	-
	1	0	2	84 / 72	78		
		4	1	68		-	-
		8	2	93 / 190 (***)	142	-	-
		16	2	90 / 103	97		
		26	1	105 (****)	-	-	-
	Overall mean, RSD and Standard deviation				**107**	**42.2**	**45.3**
Sunflower (seed)	0.01	0	1	94	-	-	-
		4	1	116	-	-	-
		8	1	90	-	-	-
		16	1	75	-	-	-
		26	1	102	-	-	-
	0.20	0	3	71 / 92 / 75	79	14.1	11.2
		4	2	68 / 78	73	-	-
		8	2	93 / 97	95	-	-
		16	2	76 / 71	73		
		26	3	84 / 86 / 74	81	7.8	6.4
	Overall mean, RSD and Standard deviation				**85**	**15.4**	**13.1**

** For dry bean, at T = 9 months, the high values obtained for the concurrent recoveries at 0.2 mg/kg were due to contamination with BYF 14182-homoglutathione of the acidified water used to dilute extract A

*** For wheat (straw), at T = 8months, the high values obtained for the concurrent recoveries at 0.05 mg/kg and 1 mg/kg were due to contamination with BYF 14182-homoglutathione of the acidified water used to dilute extract A

**** Two results were not provided as they were reported to be outside of the criteria.

Table B.7.6.2-11 Summary of concurrent recoveries of BYF 14182-pyrazole-4-carboxamide.

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-pyrazole-4-carboxamide							
Potato (tuber)	0.01	0	1	119	-	-	-
		3	1	83	-	-	-
		9	1	90	-	-	-
		17	1	92	-	-	-
		26	1	68	-	-	-
	0.10	3	2	85 / 77	81	-	-
	0.20	0	3	95 / 96 / 89	93	4.1	3.8
		9	2	99 / 104	102	-	-
		17	2	99 / 91	95	-	-
		26	3	86 / 91/ 90	89	3.0	3.5
	Overall mean, RSD and Standard deviation				**91**	**12.2**	**11.1**
Lettuce	0.01	0	1	114	-	-	-

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-pyrazole-4-carboxamide							
(head)		3	1	105	-	-	-
		9	1	105	-	-	-
		16	1	96	-	-	-
		26	1	61	-	-	-
	0.40	9	2	106 / 109	108	-	-
	0.20	0	3	92 / 87 / 86	88	3.6	3.2
		3	2	87 / 88	88	-	-
		16	2	95 / 95	95	-	-
		26	3	90 / 96 / 97	94	4.0	3.8
	Overall mean, RSD and Standard deviation				**95**	**15.6**	**14.8**
Dry bean (seed)	0.01	0	1	88	-	-	-
		3	1	109	-	-	-
		9	1	79	-	-	-
		17	1	101	-	-	-
		26		*	-	-	-
	0.20	0	3	93 / 88 / 93	91	3.2	2.9
		3	2	97 / 94	96	-	-
		9	2	105 / 104	105	-	-
		17	2	98 / 100	99	-	-
		26	3	93 / 85 / 92	90	4.8	4.4
	Overall mean, RSD and Standard deviation				**95**	**8.3**	**7.9**
Orange (fruit)	0.01	0	1	104	-	-	-
		3	1	73	-	-	-
		8	1	104	-	-	-
		16	1	109	-	-	-
		26	1	81	-	-	-
	0.20	0	3	97 / 97 / 93	96	2,4	2,3
		3	2	95 / 91	93	-	-
		8	2	99 / 101	100	-	-
		16	2	87 / 93	90	-	-
		26	3	94 / 85 / 92	91	5.1	4.7
	Overall mean, RSD and Standard deviation				**94**	**9.5**	**8.9**
Wheat (grain)	0.01	0	1	94	-	-	-
		4	1	93	-	-	-
		9	1	101	-	-	-
		16	1	88	-	-	-
		26		*	-	-	-
	0.20	0	3	97 / 98 / 94	96	2.2	2.1
		4	2	89 / 95	92	-	-
		9	2	114 / 113	114	-	-
		16	2	99 / 94	96		
		26	3	108 / 101 / 90	100	9.1	9.1
	Overall mean, RSD and Standard deviation				**98**	**8.0**	**7.9**
Wheat (straw)	0.05	0	1	88	-	-	-
		4	1	76	-	-	-
		8	1	90	-	-	-
		16	1	94	-	-	-
		26	1	72	-	-	-
	1	0	3	87 / 86 / 88	87	1.1	1.0

Matrix	Fortification level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-pyrazole-4-carboxamide							
		4	2	84 / 83	84	-	-
		8	2	94 / 95	95	-	-
		16	2	81 / 84	83	-	-
		26	3	82 / 82 / 94	86	7.7	6.9
	Overall mean, RSD and Standard deviation				**86**	**7.5**	**6.5**
Sunflower (seed)	0.01	0	1	87	-	-	-
		4	1	76	-	-	-
		8	1	110	-	-	-
		16	1	101	-	-	-
		27		*	-	-	-
	0.20	0	3	91 / 92 / 91	91	0.6	0.6
		4	2	86 / 88	87	-	-
		8	2	117 / 108	113	-	-
		16	2	104 / 101	103	-	-
		27	3	110 / 106 / 108	108	1.9	2.0
	Overall mean, RSD and Standard deviation				**99**	**11.6**	**11.4**

*Result not provided as reported to be outside criteria.

Table B.7.6.2-12 Summaries of concurrent recoveries of BYF 14182-bis-desmethyl-3-carboxylic acid.

Matrix	Spike level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-bis-desmethyl-3-carboxylic acid							
Potato (tuber)	0.01	0	1	96	-	-	-
		3	1	96	-	-	-
		9	1	90	-	-	-
		17	1	80	-	-	-
		26	1	60	-	-	-
	0.10	3	2	82 / 83	83	-	-
	0.20	0	3	85 / 85 / 82	84	2.1	1.7
		9	2	83 / 89	86	-	-
		17	2	82 / 80	81	-	-
		26	3	64 / 67 /67	66	2.6	1.7
	Overall mean, RSD and Standard deviation				**81**	**13.0**	**10.5**
Lettuce (head)	0.01	0	1	88	-	-	-
		3	1	93	-	-	-
		9	1	82	-	-	-
		16	1	95	-	-	-
		26	1	80	-	-	-
	0.40	9	2	90 / 94	92	-	-
	0.20	0	3	81 / 75 / 82	79	4.8	3.8
		3	2	85 / 86	86	-	-
		16	2	77 / 87	82	-	-
		26	3	68 / 66 / 72	69	4.4	3.1
	Overall mean, RSD and Standard deviation				**82**	**10.6**	**8.7**
Dry bean (seed)	0.01	0	1	83	-	-	-
		3	1	73	-	-	-

Matrix	Spike level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-bis-desmethyl-3-carboxylic acid							
		9	1	67	-	-	-
		17	1	94	-	-	-
		26	1	80	-	-	-
	0.20	0	3	89 / 86 / 84	86	2.9	2.5
		3	2	86 / 81	84	-	-
		9	2	88 / 86	87	-	-
		17	2	93 / 98	95	-	-
		26	3	74 / 56 / *	65	-	-
	Overall mean, RSD and Standard deviation				**82**	**13.0**	**10.7**
Orange (fruit)	0.01	0	1	93	-	-	-
		3	1	99	-	-	-
		8	1	85	-	-	-
		16	1	91	-	-	-
		26	1	86	-	-	-
	0.20	0	3	79 / 85 / 76	80	5.7	4.6
		3	2	93 / 88	91	-	-
		8	2	101 / 101	101	-	-
		16	2	78 / 91	85	-	-
		26	3	74 / 78 / 79	77	3.7	2.6
	Overall mean, RSD and Standard deviation				**87**	**10.1**	**8.8**
Wheat (grain)	0.01	0	1	93	-	-	-
		4	1	75	-	-	-
		9	1	87	-	-	-
		16	1	98	-	-	-
		26	1	87	-	-	-
	0.20	0	3	87 / 80 / 79	82	5.3	4.4
		4	2	79 / 88	84	-	-
		9	2	93 / 95	94	-	-
		16	2	92 / 87	90		
		26	3	57 / 59 / 75	64	15.5	9.9
	Overall mean, RSD and Standard deviation				**83**	**14.0**	**11.6**
Wheat (straw)	0.05	0	1	79	-	-	-
		4	1	86	-	-	-
		9	1	79	-	-	-
		16	1	84	-	-	-
		26	1	66	-	-	-
	1	0	3	76 / 75 / 80	77	3.4	2.6
		4	2	95 / 92	94	-	-
		8	2	96 / 97	97	-	-
		16	2	80 / 83	82	-	-
		26	3	65 / 76 / 69	70	7.8	5.6
	Overall mean, RSD and Standard deviation				**81**	**12.2**	**9.9**
Sunflower (seed)	0.01	0	1	83	-	-	-
		4	1	107	-	-	-
		8	1	90	-	-	-
		16	1	72	-	-	-
		26	1	91	-	-	-
	0.20	0	3	72 / 67 / 73	71	4.5	3.2
		4	2	81 / 96	89	-	-

Matrix	Spike level (mg/kg)	Storage Interval (months)	Sample size (n)	Recoveries (%)	Mean (%)	RSD (%)	Standard deviation (%)
BYF 14182-bis-desmethyl-3-carboxylic acid							
		8	2	72 / 89	81	-	-
		16	2	80 / 77	79	-	-
		26	3	94 / 92 / 92	93	1.2	1.2
	Overall mean, RSD and Standard deviation				**84**	**13.1**	**11.0**

*One result not provided as reported to be outside criteria.

Residues of BYF14182 and its metabolites in unfortified control samples were generally <0.003 mg/kg (<0.015 mg/kg for straw), Table B.7.6.2-13. An exception was the 8 month control sample for wheat straw which contained a BYF 14182-homoglutathione residue of 0.05 mg/kg which was reported to be due to contamination of the dilution solvent. Nine and 26 month dry bean samples also contained BYF 14182-homoglutathione residues of 0.01 and 0.004 mg/kg respectively.

Table B.7.6.2-13 Residues of BYF 14182 and its metabolites in stored control samples.

	Storage interval (months)	Residue (mg/kg)				
		BYF 14182	BYF 14182-hydroxy-butyl	BYF 14182-homoglutathione	BYF 14182-pyrazole-4-carboxamide	BYF 14182-bis-desmethyl-3-carboxylic acid
Potato (tuber)	0	<0.003	<0.003	0.003	<0.003	<0.003
	3	<0.003	<0.003	<0.003	<0.003	<0.003
	9	<0.003	<0.003	<0.003	<0.003	<0.003
	17	<0.003	<0.003	<0.003	<0.003	<0.003
	26	<0.003	<0.003	<0.003	<0.003	<0.003
Lettuce (head)	0	<0.003	<0.003	<0.003	<0.003	<0.003
	3	<0.003	<0.003	<0.003	<0.003	<0.003
	9	<0.003	<0.003	<0.003	<0.003	<0.003
	16	<0.003	<0.003	<0.003	<0.003	<0.003
	26	<0.003	<0.003	<0.003	<0.003	<0.003
Dry bean (seed)	0	<0.003	<0.003	<0.003	<0.003	<0.003
	3	<0.003	<0.003	<0.003	<0.003	<0.003
	9	<0.003	<0.003	0.012*	<0.003	<0.003
	17	<0.003	<0.003	<0.003	<0.003	<0.003
	26	<0.003	<0.003	0.0035	<0.003	<0.003
Orange (fruit)	0	<0.003	<0.003	<0.003	<0.003	<0.003
	3	<0.003	<0.003	<0.003	<0.003	<0.003

	8	<0.003	<0.003	<0.003	<0.003	<0.003
	16	<0.003	<0.003	<0.003	<0.003	<0.003
	26	<0.003	<0.003	<0.003	<0.003	<0.003
Wheat (grain)	0	<0.003	<0.003	<0.003	<0.003	<0.003
	4	<0.003	<0.003	<0.003	<0.003	<0.003
	9	<0.003	<0.003	<0.003	<0.003	<0.003
	16	<0.003	<0.003	<0.003	<0.003	<0.003
	26	<0.003	<0.003	<0.003	<0.003	<0.003
Wheat	0	<0.015	<0.015	<0.015	<0.015	<0.015
(straw)	4	<0.015	<0.015	<0.015	<0.015	<0.015
	8	<0.015	<0.015	0.048	<0.015	<0.015
	16	<0.015	<0.015	<0.015	<0.015	<0.015
	26	<0.015	<0.015	<0.015	<0.015	<0.015
Sunflower	0	<0.003	<0.003	<0.003	<0.003	<0.003
(seed)	4	<0.003	<0.003	<0.003	<0.003	<0.003
	8	<0.003	<0.003	<0.003	<0.003	<0.003
	16	<0.003	<0.003	<0.003	<0.003	<0.003
	26	<0.003	<0.003	<0.003	<0.003	<0.003

*In original 9 month interim report this residue was reported as BCS-AA10791 (pyrazole-4-carboxamide)

The results of the storage stability of BYF 14182 and its metabolites BYF 14182-3-hydroxy-butyl, BYF 14182-homoglutathione, BYF 14182-pyrazole-4-carboxamide and BYF 14182-bis-desmethyl-3-carboxylic acid for the different matrices are summarised in Tables B.7.6.2-14 to B.7.6.2-18.

Table B.7.6.2-14 Stability of BYF 14182 in plant matrices.

Commodity	**Fortification level (mg/kg)**	**Storage interval (months)**	**Recovered residues in stored samples (mg/kg)**	**Mean recovered residues in stored samples (mg/kg)**	**Mean concurrent recovery (%)**	**Corrected recovery (%)**
BYF 14182						
Potato (tuber)	0.2	0	0.196, 0.198, 0.193	0.196 (98%)	83	118
		3	0.151, 0.157, 0.158	0.155 (78%)	89	87
		9	0.220, 0.219, 0.220	0.220 (110%)	113	97
		17	0.105, 0.165, 0.168	0.146 (73%)	81	91
		26	0.169, 0.154, 0.160	0.161 (81%)	82	99
Lettuce	0.2	0	0.192, 0.190,	0.192	86	111

(head)			0.193	(96%)		
		3	0.135, 0.122, 0.145	0.134 (67%)	72	93
		9	0.199, 0.181, 0.177	0.186 (93%)	110	85
		16	0.129, 0.113, 0.104	0.115 (58%)	65	89
		26	0.147, 0.153, 0.151	0.150 (75%)	76	99
Dry bean (seed)	0.2	0	0.115, 0.143, 0.122	0.127 (64%)	62	103
		3	0.151, 0.145, 0.146	0.147 (74%)	75	98
		9	0.135, 0.167, 0.152	0.151 (76%)	111	68
		17	0.167, 0.177, 0.182	0.175 (88%)	78	112
		26	0.151, 0.158, 0.157	0.155 (78%)	71	109
Orange (fruit)	0.2	0	0.201, 0.175, 0.171	0.182 (91%)	89	102
		3	0.165, 0.159, 0.138	0.154 (77%)	79	97
		8	0.193, 0.200, 0.191	0.195 (97%)	97	100
		16	0.157, 0.175, 0.151	0.161 (80%)	83	96
		26	0.159, 0.141	0.150 (75%)	75	100
Wheat (grain)	0.2	0	0.140, 0.132, 0.118	0.130 (65%)	69	94
		4	0.148, 0.165, 0.159	0.157 (79%)	65	121
		9	0.188, 0.186, 0.172	0.182 (91%)	94	97
		17	0.171, 0.130, 0.142	0.148 (74%)	82	90
		26	0.164, 0.108, 0.112, 0.117, 0.153, 0.126	0.130 (65%)	75	87
Wheat (straw)	1.0	0	0.759, 0.735, 0.776	0.757 (76%)	76	100
		4	0.745, 0.725, 0.757	0.742 (75%)	79	95
		9	0.963, 0.889, 0.875	0.909 (91%)	95	96
		16	0.604, 0.793, 0.855, 0.711, 0.825, 0.558	0.724 (72%)	56	129
		26	0.651, 0.548, 0.663	0.621 (62%)	66	94
Sunflower (seed)	0.2	0	0.091, 0.125, 0.124	0.113 (57%)	48	119

		4	0.135, 0.128, 0.123	0.129 (65%)	66	98
		8	0.150, 0.146, 0.145	0.147 (73%)	99	74
		17	0.162, 0.113, 0.100	0.125 (63%)	55	114
		27	0.108, 0.100, 0.155	0.121 (61%)	60	100

Table B.7.6.2-15 Stability of BYF 14182-3-hydroxy-butyl in plant matrices.

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-3-hydroxy-butyl						
Potato (tuber)	0.2	0	0.188, 0.193, 0.186	0.189 (95%)	90	105
		3	0.187, 0.168, 0.179	0.178 (89%)	87	103
		9	0.169, 0.174, 0.177	0.173 (87%)	91	96
		17	0.172, 0.168, 0.148	0.163 (81%)	84	97
		26	0.172, 0.174, 0.171	0.172 (86%)	87	99
Lettuce (head)	0.2	0	0.190, 0.182, 0.170	0.181 (91%)	88	103
		3	0.178, 0.176, 0.173	0.176 (88%)	78	113
		9	0.168, 0.156, 0.151	0.158 (79%)	91	87
		16	0.190, 0.189, 0.199	0.193 (96%)	96	100
		26	0.173, 0.169, 0.171	0.171 (86%)	88	97
Dry bean (seed)	0.2	0	0.146, 0.161, 0.152	0.153 (77%)	77	100
		3	0.160, 0.167, 0.160	0.162 (81%)	66	123
		9	0.143, 0.155, 0.153	0.150 (75%)	98	77
		17	0.178, 0.188, 0.186	0.184 (92%)	76	121
		26	0.155, 0.155, 0.154	0.155 (77%)	75	103
Orange (fruit)	0.2	0	0.194, 0.192, 0.183	0.190 (95%)	86	110

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-3-hydroxy-butyl						
		3	0.157, 0.161, 0.161	0.160 (80%)	94	85
		8	0.159, 0.189, 0.176	0.175 (88%)	92	95
		16	0.156, 0.155, 0.177	0.163 (81%)	81	100
		26	0.170, 0.162, 0.168	0.167 (83%)	85	99
Wheat (grain)	0.2	0	0.164, 0.137, 0.173	0.158 (79%)	80	99
		4	0.152, 0.170, 0.167	0.163 (82%)	66	124
		9	0.174, 0.169, 0.203	0.182 (91%)	95	96
		16	0.173, 0.171, 0.194	0.179 (90%)	79	114
		26	0.159, 0.165, 0.165	0.163 (81%)	79	103
Wheat (straw)	1.0	0	0.952, 0.931, 0.936	0.940 (94%)	87	108
		4	0.712, 0.990, 0.796	0.833 (83%)	100	83
		9	0.894, 0.768, 0.798	0.820 (82%)	78	105
		16	0.831, 0.736, 0.873	0.813 (81%)	71	115
		26	0.795, 0.716, 0.773	0.761 (76%)	75	102
Sunflower (seed)	0.2	0	0.161, 0.188, 0.178	0.176 (88%)	81	109
		4	0.146, 0.183, 0.178	0.169 (85%)	86	98
		8	0.166, 0.158, 0.148	0.157 (79%)	94	84
		9	0.157, 0.157, 0.160	0.158 (79%)	82	96
		26	0.154, 0.151, 0.144	0.150 (75%)	83	90

Table B.7.6.2-16 Stability of BYF 14182-homoglutathione in plant matrices.

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-homoglutathione						

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-homoglutathione						
Potato (tuber)	0.2	0	0.205, 0.197, 0.185	0.196 (98%)	94	105
		3	0.216, 0.224, 0.183	0.208 (104%)	107	97
		9	0.237, 0.230, 0.228	0.232 (116%)	130	89
		17	0.216, 0.163, 0.245	0.208 (104%)	114	92
		26	0.213, 0.211, 0.206	0.210 (105%)	121	87
Lettuce (head)	0.2	0	0.158, 0.171, 0.149	0.159 (80%)	77	103
		3	0.225, 0.235	0.230 (115%)	90	128
		9	0.207, 0.225, 0.213	0.215 (108%)	118	91
		16	0.201, 0.232, 0.207	0.213 (107%)	102	105
		26	0.202, 0.181, 0.202	0.195 (98%)	105	93
Dry bean (seed)	0.2	0	0.124, 0.117, 0.114	0.118 (59%)	51	116
		3	0.216, 0.226, 0.202	0.215 (107%)	106	101
		9	0.211, 0.246, 0.208	0.222 (111%)	156	71
		17	0.241, 0.237, 0.228	0.235 (118%)	104	113
		26	0.189, 0.243, 0.238	0.223 (111%)	90	123
Orange (fruit)	0.2	0	0.141, 0.146, 0.160	0.149 (75%)	82	91
		3	0.199, 0.161, 0.218	0.193 (96%)	88	109
		8	0.191, 0.188, 0.180	0.186 (93%)	92	101
		16	0.172, 0.173, 0.172	0.172 (86%)	92	94
		26	0.153, 0.204, 0.150	0.169 (85%)	91	93
Wheat (grain)	0.2	0	0.098, 0.115, 0.095	0.103 (52%)	83	62

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-homoglutathione						
		4	0.165, 0.169, 0.178	0.171 (85%)	76	112
		9	0.155, 0.148	0.152 (76%)	82	92
		16	0.201, 0.190, 0.213	0.201 (100%)	111	90
		26	0.139, 0.140, 0.128, 0.232, 0.142, 0.143	0.154 (77%)	87	89
Wheat (straw)	1.0	0	0.807, 0.722, 0.801	0.777 (78%)	78	100
		4	0.888, 0.603, 1.000	0.830 (83%)	68	122
		9	1.340, 1.250, 1.440	1.343 (134%)	142	95
		16	1.140, 1.280, 1.210	1.210 (121%)	97	125
		26	0.862, 0.874, 0.947	0.894 (89%)	105	85
Sunflower (seed)	0.2	0	0.155, 0.137, 0.135	0.142 (71%)	79	90
		4	0.059, 0.216, 0.229	0.168 (84%)	73	115
		8	0.145, 0.137, 0.146	0.143 (72%)	95	75
		16	0.138, 0.129, 0.112	0.126 (63%)	73	86
		26	0.172, 0.153, 0.151	0.159 (79%)	81	98

Table B.7.6.2-17 Stability of BYF 14182-pyrazole-4-carboxamide in plant matrices.

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-pyrazole-4-carboxamide						
Potato (tuber)	0.2	0	0.200, 0.200, 0.202	0.201 (101%)	93	108

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-pyrazole-4-carboxamide						
		3	0.186, 0.192, 0.192	0.190 (95%)	81	117
		9	0.181, 0.176, 0.174	0.177 (89%)	102	87
		17	0.197, 0.191, 0.164	0.184 (92%)	95	97
		26	0.184, 0.197, 0.176	0.186 (93%)	89	104
Lettuce (head)	0.2	0	0.186, 0.188, 0.190	0.188 (94%)	88	107
		3	0.184, 0.187, 0.194	0.188 (94%)	88	107
		9	0.181, 0.173, 0.169	0.174 (87%)	108	81
		16	0.196, 0.193, 0.191	0.193 (97%)	95	102
		26	0.183, 0.183, 0.180	0.182 (91%)	94	97
Dry bean (seed)	0.2	0	0.186, 0.184, 0.185	0.185 (93%)	91	102
		3	0.191, 0.202, 0.197	0.197 (99%)	96	103
		9	0.168, 0.182, 0.170	0.173 (87%)	105	83
		17	0.201, 0.197, 0.200	0.199 (100%)	99	101
		26	0.190, 0.189, 0.184	0.188 (94%)	90	104
Orange (fruit)	0.2	0	0.197, 0.186, 0.195	0.193 (97%)	96	101
		3	0.188, 0.206, 0.199	0.198 (99%)	93	106
		8	0.174, 0.174, 0.170	0.173 (86%)	100	86
		16	0.193, 0.190, 0.198	0.194 (97%)	90	108
		26	0.193, 0.193, 0.183	0.190 (95%)	91	105
Wheat (grain)	0.2	0	0.179, 0.176, 0.172	0.176 (88%)	96	92
		4	0.197, 0.205, 0.200	0.201 (101%)	92	109
		9	0.181, 0.180, 0.179	0.180 (90%)	114	79
		16	0.199, 0.195, 0.199	0.198 (99%)	96	103
		26	0.197, 0.175, 0.183	0.185 (92%)	100	92
Wheat	1.0	0	0.833, 0.886,	0.847	87	97

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-pyrazole-4-carboxamide						
(straw)			0.822	(85%)		
		4	0.875, 0.851, 0.825	0.850 (85%)	84	102
		9	0.774, 0.785, 0.733	0.764 (76%)	95	80
		16	0.964, 0.866, 0.922	0.917 (92%)	83	110
		26	0.958, 0.837, 0.831	0.875 (88%)	86	102
Sunflower (seed)	0.2	0	0.184, 0.179, 0.180	0.180 (90%)	91	99
		4	0.188, 0.192, 0.177	0.186 (93%)	87	107
		8	0.196, 0.195, 0.196	0.196 (98%)	113	86
		16	0.225, 0.212, 0.233	0.223 (112%)	103	109
		27	0.237, 0.200, 0.212	0.216 (108%)	108	100

Table B.7.6.2-18 Stability of BYF 14182-bis-desmethyl-3-carboxylic acid in plant matrices.

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-bis-desmethyl-3-carboxylic acid						
Potato (tuber)	0.2	0	0.178, 0.171, 0.186	0.178 (89%)	84	106
		3	0.174, 0.159, 0.173	0.169 (85%)	83	102
		9	0.174, 0.178, 0.173	0.175 (88%)	86	102
		17	0.176, 0.183, 0.176	0.178 (89%)	81	110
		26	0.139, 0.123, 0.134	0.132 (66%)	66	100
Lettuce (head)	0.2	0	0.164, 0.171, 0.169	0.168 (84%)	79	107

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-bis-desmethyl-3-carboxylic acid						
		3	0.192, 0.186, 0.181	0.186 (93%)	86	109
		9	0.173, 0.168, 0.164	0.168 (84%)	92	92
		16	0.163, 0.187, 0.187	0.179 (90%)	82	110
		26	0.149, 0.141, 0.137	0.142 (71%)	69	104
Dry bean (seed)	0.2	0	0.177, 0.181, 0.183	0.180 (90%)	86	105
		3	0.172, 0.180, 0.158	0.170 (85%)	84	101
		9	0.150, 0.156, 0.158	0.155 (78%)	87	89
		17	0.206, 0.214, 0.191	0.204 (102%)	95	107
		26	0.145, 0.118, 0.128	0.130 (65%)	65	100
Orange (fruit)	0.2	0	0.146, 0.144, 0.141	0.144 (72%)	80	90
		3	0.190, 0.184, 0.187	0.187 (94%)	91	103
		8	0.188, 0.188, 0.187	0.188 (94%)	101	93
		16	0.202, 0.212, 0.210	0.208 (104%)	85	122
		26	0.188, 0.160, 0.157	0.168 (84%)	77	110
Wheat (grain)	0.2	0	0.158, 0.156, 0.162	0.159 (80%)	82	97
		4	0.157, 0.172, 0.178	0.169 (85%)	84	101
		9	0.177, 0.168, 0.169	0.171 (86%)	94	91
		16	0.196, 0.193, 0.203	0.197 (99%)	90	110
		26	0.157, 0.120, 0.147	0.141 (71%)	64	110
Wheat (straw)	1.0	0	0.813, 0.746, 0.761	0.773 (77%)	77	100
		4	0.893, 0.970, 0.923	0.929 (93%)	94	99
		9	0.912, 0.909, 0.867	0.896 (90%)	97	92
		16	0.862, 0.896, 0.972	0.910 (91%)	82	111
		26	0.864, 0.824, 0.723	0.804 (80%)	70	114
Sunflower	0.2	0	0.159, 0.140,	0.139	71	99

Commodity	Fortification level (mg/kg)	Storage interval (months)	Recovered residues in stored samples (mg/kg)	Mean recovered residues in stored samples (mg/kg)	Mean concurrent recovery (%)	Corrected recovery (%)
BYF 14182-bis-desmethyl-3-carboxylic acid						
(seed)			0.119	(70%)		
		4	0.199, 0.172, 0.221	0.197 (99%)	89	111
		8	0.163, 0.218, 0.177	0.186 (93%)	81	115
		16	0.218, 0.202, 0.178	0.199 (100%)	79	126
		26	0.202, 0.186, 0.192	0.193 (97%)	93	104

Potato (tuber)

After 26 months storage under deep frozen conditions the average corrected recoveries of BYF 14182 and its metabolites were 99% for BYF 14182, 99% for BYF 14182-3-hydroxy-butyl, 87% for BYF 14182-homoglutathione, 104% for BYF 14182-pyrazole-4-carboxamide and 100% for BYF 14182-bis-desmethyl-3-carboxylic acid. BYF 14182 and its metabolites were therefore shown to be stable in potato tubers when stored frozen for at least 26 months.

Lettuce (head)

After 26 months storage under deep frozen conditions the average corrected recoveries of BYF 14182 and its metabolites were 99% for BYF 14182, 97% for BYF 14182-3-hydroxy-butyl, 93% for BYF 14182-homoglutathione, 97% for BYF 14182-pyrazole-4-carboxamide and 104% for BYF 14182-bis-desmethyl-3-carboxylic acid. BYF 14182 and its metabolites were therefore shown to be stable in lettuce head when stored frozen for at least 26 months.

Dry bean (seed)

After 26 months storage under deep frozen conditions the average corrected recoveries of BYF 14182 and its metabolites were 109% for BYF 14182, 103% for BYF 14182-3-hydroxy-butyl, 123% for BYF 14182-homoglutathione, 104% for BYF 14182-pyrazole-4-carboxamide and 100% for BYF 14182-bis-desmethyl-3-carboxylic acid. BYF 14182 and its metabolites were therefore shown to be stable in dry bean (seed) when stored frozen for at least 26 months.

Orange (fruit)

After 26 months storage under deep frozen conditions the average corrected recoveries of BYF 14182 and its metabolites were 100% for BYF 14182, 99% for BYF 14182-3-hydroxy-butyl, 93% for BYF 14182-homoglutathione, 105% for BYF 14182-pyrazole-4-carboxamide and 110% for BYF 14182-bis-desmethyl-3-

carboxylic acid. BYF 14182 and its metabolites were therefore shown to be stable in orange fruit when stored frozen for at least 26 months.

Wheat (grain)

After 26 months storage under deep frozen conditions the average corrected recoveries of BYF 14182 and its metabolites were 87% for BYF 14182, 103% for BYF 14182-3-hydroxy-butyl, 89% for BYF 14182-homoglutathione, 92% for BYF 14182-pyrazole-4-carboxamide and 110% for BYF 14182-bis-desmethyl-3-carboxylic acid. BYF 14182 and its metabolites were therefore shown to be stable in wheat grain when stored frozen for at least 26 months.

Wheat (straw)

After 26 months storage under deep frozen conditions the average corrected recoveries of BYF 14182 and its metabolites were 94% for BYF 14182, 102% for BYF 14182-3-hydroxy-butyl, 85% for BYF 14182-homoglutathione, 102% for BYF 14182-pyrazole-4-carboxamide and 114% for BYF 14182-bis-desmethyl-3-carboxylic acid. BYF 14182 and its metabolites were therefore shown to be stable in wheat straw when stored frozen for at least 26 months.

Sunflower (seed)

After 26-27 months storage under deep frozen conditions the average corrected recoveries of BYF 14182 and its metabolites were 100% for BYF 14182, 90% for BYF 14182-3-hydroxy-butyl, 98% for BYF 14182-homoglutathione, 100% for BYF 14182-pyrazole-4-carboxamide and 104% for BYF 14182-bis desmethyl-3-carboxylic acid. BYF 14182 and its metabolites were therefore shown to be stable in sunflower seed when stored frozen for at least 26 months.

Conclusions

While the recoveries from the storage stability samples were variable, the variability was also reflected in the concurrent recovery experiments. The corrected recoveries in the storage stability study suggest BYF 14182 and its metabolites are stable in water, starch, protein, oil and acid containing materials for up to 26-27 months when stored frozen.

Possible degradation of parent and BYF 14182-homoglutathione in dry bean were noted in the initial evaluation of the study for up to 9 months storage, however the 17 month interim report appeared to show acceptable recoveries at this time point. This is confirmed now that the final study report is available. Issues with storage stability recoveries of parent from sunflower seed appear to be related to problems with the method. The storage stability of parent in sunflower seed is acceptable if corrected for concurrent recoveries.